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| <p>2002-124420/17 B05 C03 RHOD 2000.06.21 RHODIA CHIM *FR 2810665-A1 2000.06.21 2000-007924(+2000FR-007924) (2001.12.28) C07C 211/46, C07D 231/12 Preparation of aniline having chlorine atom and perhalogenated aliphatic group with fluorine atom, used as intermediates for e.g. pharmaceuticals, by contacting N-aryl carbamoyl fluoride with chlorinating agent in hydrofluoric medium C2002-038307 Addnl. Data: SAINT JALMES L, SCHANEN V</p> | <p>B(10-B4A, 11-C1) C(10-B4A, 11-C1) .2</p> <p>includes the stage of preparing (I) as above.</p> <p><u>USE</u> Used for preparation of intermediates for pharmaceutical and agrochemical products, especially intermediates for the preparation of Fipronil.</p> |
| <p><u>NOVELTY</u> Preparation of an aniline (I) having both a chlorine atom and a perhalogenated aliphatic group with at least one fluorine atom, on the aromatic ring, comprises contacting, at at least 50°C, a N-aryl carbamoyl fluoride with a chlorinating agent in a hydrofluoric medium capable of exchanging the chlorine and the fluorine on the benzyl position or on a carbon carrying a chalcogen.</p> <p><u>DETAILED DESCRIPTION</u> An INDEPENDENT CLAIM is included for the preparation of an N-phenylpyrazole dichlorinated on the ortho-ortho'-position of the pyrazole and having an electro-tractor on the para position, which</p> | <p><u>ADVANTAGE</u> The process is easier to carry out, and produces less saline waste material, than known methods.</p> <p><u>EXAMPLE</u> A reaction mixture from the fluorination of 4-trichloromethylphenyl isocyanate, containing 1 mole of carbamoyl 4-trifluoromethyl phenyl fluoride and 5.5 moles of hydrofluoric acid, was treated in a closed autoclave, with 2.2 molar equivalents of chlorine. The mixture was heated to 90-110°C for 2-3 hours, after which analysis showed that most of the aromatic compounds present had a carbamoyl fluoride and/or isocyanate group dichlorinated on the</p> <p>FR 2810665-A+</p> |

aromatic ring, together with traces of 2,6-dichloro-4-trifluoromethyl aniline. The mixture was cooled and neutralized, and the solvent evaporated to give an oil (225 g) most of which was 2,6-dichloro-4-trifluoromethyl aniline.

TECHNOLOGY FOCUS

Organic Chemistry - Preferred Process: Chlorination is effected by bubbling chlorine into the mixture at atmospheric pressure or above and at a temperature of 70-120°C. The mixture is then rapidly cooled and hydrofluoric acid eliminated under reduced pressure.
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